JC03 Rec'd 30 OCT 2001

FORM PTO-1390 (REV 12-97) U S DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE

DATE: OCTOBER 30 2001

TRANSMITTAL LETTER TO THE UNITED STATES DESIGNATED/ELECTED OFFICE (DO/EO/US) CONCERNING A FILING UNDER 35 U.S.C. 371

ATTORNEY'S DOCKET NUMBER: 54779US005

U.S. APPLICATION NO. (if known, see 37 C.F.R. 1.5): 09/980674

INTERNATIONAL APPLICATION NO.	INTERNATIONAL FILING DATE	PRIORITY DATE CLAIMED
PCT/US00/11422	27 April 2000	7 June 1999

TITLE OF INVENTION
ADHESIVE COMPOSITION AND OPTICAL DISC USING THE SAME

APPLICANT(S) FOR DO/EO/US
FUJII, Sana

Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:

1. ☒ This is a FIRST submission of items concerning a filing under 35 U.S.C. 371.
2. ☐ This is a SECOND or SUBSEQUENT submission of items concerning a filing under 35 U.S.C. 371.
3. ☐ This express request to begin national examination procedures (35 U.S.C. 371(f)) at any time rather than delay examination until the expiration of the applicable time limit set in 35 U.S.C. 371(b) and PCT Articles 22 and 39 (1).
4. ☒ A proper Demand for International Preliminary Examination was made by the 19th month from the earliest claimed priority date.
5. ☒ A copy of the International Application as filed (35 U.S.C. 371(c)(2)).
   - ☐ is transmitted herewith (required only if not transmitted by the International Bureau).
   - ☐ has been transmitted by the International Bureau.
   - ☒ is not required, as the application was filed in the United States Receiving Office (RO/US)
6. ☐ A translation of the International Application into English (35 U.S.C. 371(c)(2)).
   - ☐ is attached hereto
   - ☐ Has been previously submitted under 35 U.S.C. 154(d)(4)
7. ☒ Amendment to the claims of the International Application under PCT Article 19 (35 U.S.C. 371(c)(3))
   - ☐ are transmitted herewith (required only if not transmitted by the International Bureau).
   - ☐ have been transmitted by the International Bureau.
   - ☐ have not been made; however, the time limit for making such amendments has NOT expired.
   - ☒ have not been made and will not be made.
8. ☐ A translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3)).
9. ☒ An oath or declaration of the inventor(s) (35 U.S.C. 371 (c)(4)).
10. ☐ A translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(c)(5)).

Items 11. to 16. below concern other document(s) or information included:

11. ☐ An Information Disclosure Statement under 37 CFR 1.97 and 1.98.
12. ☒ An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.
13. ☒ A FIRST preliminary amendment.

    ☐ A SECOND or SUBSEQUENT preliminary amendment.
14. ☐ A substitute specification.
15. ☐ A change of power of attorney and/or address letter.
16. ☐ Other items or information:

Certificate of Express Mailing

Under 37 CFR 1.10, I certify that this entry of a PCT application into US National Stage is being deposited with the United States Postal Service "Express Mail Post Office to Addressee" service addressed to the Commissioner for Patents, Washington, DC 20231, on the date indicated below.

Express Mail Mailing Label No.	Signature of Person Mailing Application
EL596980291US	Kathleen M. Goers
Date of Deposit: OCTOBER 30 2001	Printed Name of Person Mailing Application: Kathleen M. Goers

JC10 Rec'd PCT/PTO 30 OCT 2001

U.S. APPLICATION NO. (if known, see 37 C.F.R. 1.5) 09/980674	INTERNATIONAL APPLICATION NO.:PCT/US00/11422	ATTORNEY'S DOCKET NUMBER 54779US005

17. ☒ The following fees are submitted:

Basic National Fee (37 CFR 1.492(a)(1)-(5)):

		Calculations	PTO Use Only
☒	Search Report has been prepared by the EPO or JPO $890		
☐	International preliminary examination fee paid to USPTO (37 CFR 1.482) ...$710		
☐	No international preliminary examination fee paid to USPTO (37 CFR 1.482) but international search fee paid to USPTO (37 CFR 1.445 (a)(2)) $740		
☐	Neither international preliminary examination fee (37 CFR 1.482) nor international search fee (37 CFR 1.445(a)(2)) paid to USPTO $1,040		
☐	International preliminary examination fee paid to USPTO (37 CFR 1.482) and all claims satisfied provisions of PCT Article 33(2)-(4) $100		
	ENTER APPROPRIATE BASIC FEE AMOUNT =	$ 890.	
	Surcharge of $ 130 for furnishing the oath or declaration later than ☐ 20 ☐ 30 months from the earliest claimed priority date (37 CFR 1.492(e)).	$	

Claims	Number Filed	Number Extra	Rate		
Total Claims	**6 -20 =**	**= 0**	**x $ 18**	$ 0.	
Independent Claims	**2 -3=**	= 0	**x $ 84**	$ 0.	
MULTIPLE DEPENDENT CLAIM(S) (IF APPLICABLE)			**x $ 280**	$ 0.	
			TOTAL OF ABOVE CALCULATION =	$ 890.	
Reduction by ½ for filing by small entity, if applicable. A Small Entity Statement must also be filed. (Note 37 CFR 1.9, 1.27, 1.28)				$	
			SUB TOTAL =	$ 890.	
Processing fee of $ 130 for furnishing the English translation later the ☐ **20** ☐ **30 months from the earliest priority date (37 CFR 1.492(f)).**				$ 0.	
			TOTAL NATIONAL FEE =	$ 890.	
Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be **accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31). $40.00 per property**				$	
			TOTAL FEES ENCLOSED =	$	
				Amount to be refunded	$
				charged	$ 890.

a. ☐ A check in the amount of $ _____ to cover the above fees is enclosed.

b. ☒ Please charge my Deposit Account No. 13-3723 in the amount of $ 890.00 to cover the above fees. A duplicate copy of this sheet is enclosed.

c. ☒ The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit Account No. 13-3723 A duplicate copy of this sheet is enclosed

NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137(a) or (b)) must be filed and granted to restore the application to pending status.

Send All Correspondence To:

Signature Scott A. Bardell

Name: **Scott A. Bardell**

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Patent
Docket No: 54779US005

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of:
FUJII, SANA

Group Art Unit:

International
Application No.: PCT/US00/11422

International
Filing Date: 27 April 2000

Examiner:

For: ADHESIVE COMPOSITION AND OPTICAL DISC USING THE SAME

PRELIMINARY AMENDMENT

Commissioner for Patents
Washington, DC 20231

Dear Sir:

Please enter the following preliminary amendment in the above-referenced application.

In the Claims:

Please amend claims 3, 4, and 6 as follows:

3. (Amended) The adhesive composition of claim 1, wherein the acrylic ester (2) is one or more of hydroxyethyl acrylate, hydroxypropyl acrylate, hydroxybutyl acrylate, and hydroxyphenoxypropyl acrylate.

4. (Amended) An optical disc comprising disc substrate bonded by an adhesive and at least one reflecting metallic film, characterized in that the adhesive is obtained by cure of the adhesive composition of claim 1.

6. (Amended) The adhesive composition of claim 1 wherein the ratio of poly(tetramethylene glycol), diisocyante, and linear or branched acrylic ester is about 62:15:14 parts by weight respectively.

A version marked up to show changes made to the claim(s) relative to the previous version of the claim(s) is attached.

Following is a clean version of the entire set of pending claims:

1. An adhesive composition comprising (1) a urethane acrylate having a poly(tetramethylene glycol) skeleton and comprising the reaction product of poly(tetramethylene glycol), diisocyanate, and linear or branched acrylic ester, (2) an acrylic ester having a hydroxyl group in its molecule, and (3) a photoinitiator, characterized in that the composition comprises 40% by weight or more of the urethane acrylate (1) based on the sum of weights of the urethane acrylate (1) and the acrylic ester (2).

2. The adhesive composition of claim 1, wherein the urethane acrylate (1) is an oligomer comprising the reaction product of poly (tetramethylene glycol), tetramethylxylene diisocyanate, linear or branched acrylic ester, and ethylene glycol.

3. The adhesive composition of claim 1, wherein the acrylic ester (2) is one or more of hydroxyethyl acrylate, hydroxypropyl acrylate, hydroxybutyl acrylate, and hydroxyphenoxypropyl acrylate.

4. An optical disc comprising disc substrate bonded by an adhesive and at least one reflecting metallic film, characterized in that the adhesive is obtained by cure of the adhesive composition of claim 1.

5. A method of making a urethane adhesive having a poly(tetramethylene glycol) skeleton moiety comprising the step of:

reacting a urethane acrylate comprising the reaction product of poly(tetramethylene glycol), diisocyanate, and linear or branched acrylic ester with (2) an acrylic ester having a hydroxyl group in its molecule, and (3) a photoinitiator, wherein the composition comprises 40% by weight or more of the urethane

acrylate (1) based on the sum of weights of the urethane acrylate (1) and the acrylic ester (2).

6. The adhesive composition of claim 1 wherein the ratio of poly(tetramethylene glycol), diisocyante, and linear or branched acrylic ester is about 62:15:14 parts by weight respectively.

Examination and consideration of the application as amended is requested.

Respectfully submitted,

By _____
Scott A. Bardell

Registration Number	Telephone Number
39,594	651/736-6935
Date 30 October, 2002	

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Version With Markings to Show Changes Made

3. (Amended) The adhesive composition of claim 1 **[or 2]**, wherein the acrylic ester (2) is one or more of hydroxyethyl acrylate, hydroxypropyl acrylate, hydroxybutyl acrylate, and hydroxyphenoxypropyl acrylate.

4. (Amended) An optical disc comprising disc substrate bonded by an adhesive and at least one reflecting metallic film, characterized in that the adhesive is obtained by cure of the adhesive composition of **[any one of claims 1 to 3]** **claim 1**.

6. (Amended) The adhesive composition of claim 1 **[or claim 2]** wherein the ratio of poly(tetramethylene glycol), diisocyante, and linear or branched acrylic ester is about 62:15:14 parts by weight respectively.

ADHESIVE COMPOSITION AND OPTICAL DISC USING THE SAME

The present invention relates to an adhesive composition for an optical disc, and more particularly to an adhesive composition useful in the manufacture of an optical disc used as an optical storage medium obtained by laminating transparent substrates and provided with at least one reflecting metallic film, as in a DVD (digital video disc or digital versatile disc).

CDs (compact discs), DVDs, and other optical discs are used extensively as recording media in a wide variety of fields, from audio and video recordings to computer-related storage. With such optical discs, digital signals recorded on the disc are sensed as variations in the reflection intensity of semiconductor laser light emitted by a read/write optical head, and the recorded data is read. Such discs can be read-only, recordable/nonerasable, or both recordable and erasable.

These optical discs are manufactured by a process in which an aluminum or other very thin (several tens of nanometers) metallic film layer is formed a reflecting layer by vapor deposition or sputtering on the surface (signal-recording surface) of a polycarbonate or other transparent substrates provided with pits that correspond to the recorded digital signal. In the case of a CD, the signal surface is further provided with a protective layer. In a DVD structure, on the other hand, two components, each of which is half the thickness of the CD substrate, are used as such transparent substrates, and these are laminated such that their signal-recording surfaces face inward. With such a DVD, the signal can be recorded on only one of the two laminated substrates, or it can be recorded on both substrates. Another feature of DVDs is that two recording layers can be provided to a single substrate because recorded signals from two signal-recording surfaces can be read from one side by moving the object lens of the optical head for reading recorded signals in the focusing direction (in the direction of propagation of laser light), in which case the reflecting film on the recording surface of the first layer (layer on which the readout laser light impinges first) is formed from gold or another semitransparent material. Consequently, DVDs employ the following systems for reading recording signals: those in which a single layer is read on one side, those in which two layers are read from each side,

those in which two layers are read from one side, and those in which each of the two layers are read from each side.

Aluminum or other metallic films formed on the signal surface of an optical disc substrate are affected by the oxygen and moisture contained in the air, and the characteristics of the films tend to deteriorate markedly. Specifically, an aluminum film affected by oxygen or contained moisture is gradually corroded and rendered transparent beginning at the disc edge, ultimately losing its reflecting characteristics. Thermoplastic resins or resins based on acrylate resins or epoxy resins have therefore been conventionally used as protective coating agents for metallic films. Epoxy ultraviolet-curing resin compositions used for this purpose are described in Japanese Patent Laid-open 2-107630, and acrylate compositions are described in Japanese Patent Laid-open 4-264166. Using such protective coating agents complicates the manufacturing process because an adhesive is applied and substrates are laminated after a protective coating has been formed on the signal surface of an optical disc substrate. Another drawback is that conventional discs are difficult to adapt to the need for higher density and minimal pit width.

Adhesives that allow substrates provided with signal surfaces to be directly bonded without the use of such protective coatings and that allow optical discs with minimal aluminum deterioration to be obtained are described, for example, in Japanese Patents Laid-open 59-71317, 63-167442, 9-31416, 10-8018, 10-102007, 10-67977, and 10-130602.

The adhesive described in Japanese Patent Laid-open 59-71317 is a photosetting adhesive composition containing a prescribed hydrocarbon compound that has two or more acryl or methacryl groups. This composition affords better adhesive power or the like between the substrates of an optical disc.

The adhesive described in Japanese Patent Laid-open 63-167442 is a composition consisting of hydrocarbon polyol-based polyurethane. Imparting flexibility to the composition *per se* yields an adhesive that can change its shape in conformity with deformation (warping) of the optical disc substrates.

The adhesive described in Japanese Patent Laid-open 9-31416 is a photosetting adhesive composition containing as its essential components an ultraviolet-curing

compound and a photopolymerization initiator whose molar absorption coefficient at wavelengths of 360–450 nm is 400 or higher.

The adhesive described in Japanese Patent Laid-open 10-8018 is an adhesive composition containing an ultraviolet-curing resin and a photopolymerization initiator obtained by combining a photopolymerization initiator whose maximum molar absorption coefficient at wavelengths of 360 nm or lower is 5000 or higher, and a photopolymerization initiator whose maximum molar absorption coefficient at wavelengths of 360 nm or higher is 500 or higher. The resulting cured products have adequate adhesive power and the like.

The adhesive described in Japanese Patent Laid-open 10-102007 is a composition having at least a prescribed bisacylphosphine oxide compound and a prescribed acylphosphine oxide compound.

A common feature of the adhesives disclosed above is that they are directly applied to optical disc substrates and are used to bond the substrates together. There is, however, no particular provision concerning their corrosion of metallic films.

The adhesive described in Japanese Patent Laid-open 10-67977 is disadvantageous in that a very expensive fluorene-skeleton acrylate is used as the starting material, that the resulting cured product has low impact resistance at room temperature because of the extremely high glass transition temperature (T_g) thereof, and the like. In addition, the durability evaluation conditions for the adhesive described in this official publication are comparatively mild (70 °C, 50% relative humidity), and the time period is short (96 hours), rendering such a durability evaluation inadequate.

The adhesive composition described in Japanese Patent Laid-open 10-130602 contains a mixture of compounds containing unsaturated groups (A), an organic peroxide (B), and a curing promoter (C) as essential components, and a photopolymerization initiator (D) as an optional component. It is reported that no visible pinholes or other defects form in vapor-deposited aluminum films when the aforementioned adhesive composition is allowed to stand for 100 hours at 80 °C and 85% relative humidity. In this case, however, the evaluation time is too short (100 hours) to yield a reliable durability evaluation, and the adhesive is difficult to use in the sense that low productivity results, the adhesive layer cannot maintain its thickness, and other problems occur because the curing rate tends to vary depending on the particular

combination of organic peroxide and curing promoter, and because curing tends to proceed rapidly during mixing or application when the curing rate is too high, and a considerable post-lamination standing time is needed when the curing rate is low.

An adhesive used for bonding optical disc substrates is generally required to have adequate adhesion characteristics, physical characteristics (particularly transparency, impact characteristics, and applicability to substrates), and curing characteristics during bonding. The adhesive is also required to have the quality of not producing, or resisting production of, corrosion of a metallic film during an environmental resistance test (durability test) when the adhesive is used for direct application to a recording surface provided with an aluminum or other metallic film. Specifically, an adhesive for bonding optical disc substrates must have the following characteristics:

(1) Adhesion Characteristics

Prescribed adhesive power must be achieved with respect to the surface of polycarbonate, aluminum, gold, or another material constituting an optical disc with a cured film thickness of 1–100 μm, and preferably about 50 μm.

(2) Impact Characteristics

Disc substrates must not separate from each other in a drop test.

(3) Adhesive Curing Characteristics

A photosetting adhesive must be able to bond substrates under exposure to ultraviolet light lasting no more than 1 minute, and preferably 1–30 seconds.

(4) Transparency

Adequate transparency to laser light (for example, red semiconductor laser light with a wavelength of 650–780 nm) must be afforded.

(5) Corrosion of Metallic Films

It is inadmissible for aluminum films with a thickness of approximately several tens of nanometers (for example, about 50 nm) to be corroded. In particular, no corrosion is permissible in an aluminum film over a period of 500 hours or longer during a durability test in an 80 °C environment with 90% relative humidity.

(6) Applicability to Disc Substrates

Must have a viscosity of 500–10,000 mPa · s, and preferably 1000–5000 mPa · s, at room temperature in the absence of a solvent, and form a cured film of

about 1–100 μm when directly applied by spin coating to the surface of a disc substrate provided with a metal layer.

Adhesives for bonding optical disc substrates that would have all these required characteristics have yet to be developed, and conventional adhesives still have problems in terms of corrosion of metallic films, impact characteristics, curing treatment conditions during bonding, or the like.

For example, the adhesives described in Japanese Patents Laid-open 10-67977 and 10-130602 are tested only briefly in respect of corrosion of aluminum films in durability tests (and, in the case of Japanese Patent Laid-open 10-67977, the testing conditions *per se* are extremely mild), and are not adequate for durability evaluation purposes. The adhesive described in Japanese Patent Laid-open 10-67977 has poor impact resistance at room temperature because of the extremely high glass transition temperature (T_g) of the cured product, and the adhesive described in Japanese Patent Laid-open 10-130602 has poor adjustability of the curing rate.

In particular, none of the optical disc adhesives currently available on the market can be regarded as adhesives that remain non-corrosive toward aluminum films over a period of 500 hours or longer during an environmental resistance test in an 80 °C environment with 90% relative humidity.

Thus, none of the conventional adhesives used for direct bonding of optical disc substrates devoid of protective coating layers for the reflecting metallic film has all the required characteristics. In particular, improvements can still be made concerning corrosivity in relation to reflecting metallic films such as aluminum films.

With the foregoing in view, it is an object of the present invention to provide an adhesive composition which is useful for direct bonding of optical disc substrates whose reflecting metallic films are devoid of protective coating layers, and which can control corrosion of a metallic film, particularly an aluminum film, in an extended durability test of a product optical disc under elevated temperature and high humidity conditions. It is also an object of the present invention to provide an optical disc having controlled corrosion of a metallic film under elevated temperature and high humidity, obtained by laminating substrates with the aid of such an adhesive composition.

The adhesive composition of the present invention comprises (1) a urethane acrylate having a poly (tetramethylene glycol) skeleton, (2) an acrylic ester having a

hydroxyl group in its molecule, and (3) a photoinitiator, characterized in that the composition comprises 40% by weight or more of the urethane acrylate (1) based on the sum of weights of the urethane acrylate (1) and the acrylic ester (2).

The optical disc of the present invention is an optical disc comprising disc substrates laminated using an adhesive, and at least one reflecting metallic film, wherein the adhesive is an adhesive composition comprising (1) a urethane acrylate having a poly (tetramethylene glycol) skeleton, (2) an acrylic ester having a hydroxyl group in its molecule, and (3) a photoinitiator, characterized by being obtained by the curing of an adhesive composition comprising 40% by weight or more of the urethane acrylate (1) based on the sum of weights of the urethane acrylate (1) and the acrylic ester (2).

Preferably, the urethane acrylate (1) is an oligomer made up of, in addition to the poly (tetramethylene glycol) skeleton moiety, at least moieties derived from tetramethylxylene diisocyanate, hydroxyethyl acrylate, and ethylene glycol.

It is also preferable for the acrylic ester (2) to be one or more of hydroxyethyl acrylate, hydroxypropyl acrylate, hydroxybutyl acrylate, and hydroxyphenoxypropyl acrylate.

Figure 1 illustrates an optical disc pertaining to one aspect of the present invention.

Figure 2 illustrates an optical disc pertaining to another aspect of the present invention.

Key to Numbers

10, 20..... optical discs

11, 11', 21, 21'..... disc substrates

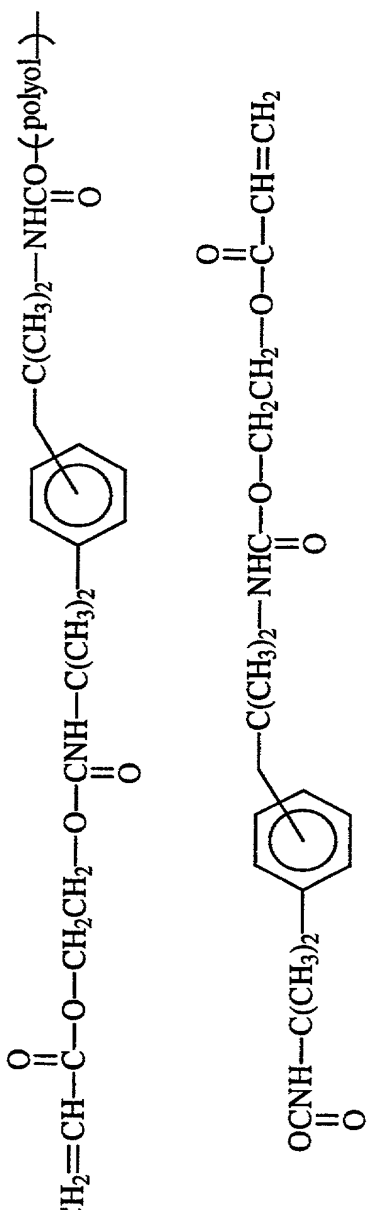
12, 24..... reflecting metallic films

13, 25..... adhesive layers

22..... semitransparent film

The principal component of the adhesive composition of the present invention is a urethane acrylate having a poly (tetramethylene glycol) skeleton. An oligomer prepared at least from tetramethylxylene diisocyanate, hydroxyethyl acrylate, and ethylene glycol components in addition to the poly (tetramethylene glycol) skeleton moiety should preferably be used as the urethane acrylate contained as the principal component in adhesive compositions of the present invention useful for controlling the corrosion of a reflecting metallic film, and an aluminum film in particular, in a durability test performed

under elevated temperature and high humidity conditions for applications involving lamination of optical disc substrates. Oligomers whose weight-average molecular weight is about 4000–7000 and the mixing molar ratio of the poly (tetramethylene glycol), tetramethylxylene diisocyanate, hydroxyethyl acrylate, and ethylene glycol is about 62:15:14:9 may be cited as examples of the urethane acrylates suitable for use in the present invention. Such urethane acrylate oligomers are commercially available from Nihon Gosei Kagaku under the trade name UV-6100B, and can be expressed by the following general formula:



The molecular weight of such a urethane acrylate oligomer and the mixing ratios of the constituent components of the oligomer may be varied broadly as long as the above-described characteristics required of the optical disc are attained.

In the adhesive composition of the present invention, an acrylic ester having a hydroxyl group in its molecule and acting as a reactive diluent is used in addition to the principal component (urethane acrylate having a poly (tetramethylene glycol) skeleton). Examples of appropriate acrylic esters include hydroxyethyl acrylate, 2-hydroxypropyl acrylate, 4-hydroxybutyl acrylate, and 2-hydroxy-3-phenoxypropyl acrylate. Hydroxyethyl acrylate, 2-hydroxypropyl acrylate, and 4-hydroxybutyl acrylate are even more preferred as acrylic esters. The reason that these are preferred is that they yield uniform compositions during mixing when the adhesive composition of the present invention is prepared, and that the viscosity of the composition can be readily adjusted.

In the adhesive composition of the present invention, the principal component (urethane acrylate having a poly (tetramethylene glycol) skeleton) must be contained in an amount of 40% by weight or more, based on the sum of weights of the urethane acrylate and the acrylic ester having a hydroxyl group in its molecule. At less than 40% by weight, the product cannot endure for 500 hours or longer in a durability test at 80 °C and 90% relative humidity. It is unsuitable for the urethane acrylate having a poly (tetramethylene glycol) skeleton to be present in an amount of more than 90% by weight, based on the sum of weights of the urethane acrylate and the acrylic ester having a hydroxyl group in its molecule, because in this case excessively high viscosity results, application is impaired, and adhesion to metallic films is compromised. A more preferred content of the urethane acrylate having a poly (tetramethylene glycol) skeleton is 50–80% by weight, based on the sum of weights of the urethane acrylate and the acrylic ester having a hydroxyl group in its molecule.

The adhesive composition of the present invention can be cured and formed into an adhesive layer on an optical disc by performing photopolymerization with the aid of a photopolymerization initiator or by performing thermopolymerization with the aid of a thermopolymerization initiator. Unlike thermopolymerization, photopolymerization-induced curing makes it possible to prevent disc substrates from developing deformation (mainly warping) and other heat-induced defects during curing. Consequently, it is preferable to perform curing based on photopolymerization. Commercially available

products may commonly be used as photopolymerization initiators. Darocur 1173 manufactured by Ciba-Geigy can be cited as a typical example of such a commercially available photopolymerization initiator.

An aspect may also be considered in which the adhesive composition of the present invention is used as a second-generation acrylic adhesive (SGA), in which case organic peroxides and curing promoters are used. In an SGA, a mixture of urethane acrylate (1) and acrylic ester (2) are divided into two components, one a component (referred to hereinbelow as "liquid A") containing an organic peroxide, and the other a component (designated as "liquid B") containing a curing promoter, which two are mixed together for use as an adhesive. Alternatively, either of the aforementioned components (for example, liquid A) may be applied to one of the disc substrates, the other component (for example, liquid B) applied to the other disc substrate, and the disc substrates then laminated together.

The adhesive composition of the present invention may also contain optional thickeners, plasticizers, dispersants, flame retardants, fillers, colorants, antioxidants, thermal aging inhibitors, UV absorbers, silane coupling agents, and various other additives commonly used in adhesive compositions.

The adhesive composition of the present invention can be easily prepared by uniformly mixing the following mandatory components with the aid of a common mixing means: a urethane acrylate having a poly (tetramethylene glycol) skeleton, an acrylic ester having a hydroxyl group in its molecule, and a photopolymerization initiator, as well as any other optional components, added as needed.

The adhesive composition should preferably be applied to an optical disc substrate by spin coating. To use spin coating, the viscosity of the coating solution should be adjusted to about 500–10,000 mPa · s, and preferably about 1000–5000 mPa · s. When the aforementioned UV-6100B available from The Nippon Synthetic Chemical Ind. Co., Ltd. or an equivalent product is used as the urethane acrylate (1) having a poly (tetramethylene glycol) skeleton, using hydroxyethyl acrylate, 2-hydroxypropyl acrylate, 4-hydroxybutyl acrylate, or a mixture thereof as the acrylic ester (2) having a hydroxyl group in its molecule can make it easier to obtain a viscosity that falls within the appropriate range. Application of the adhesive composition is not limited to spin coating and may be accomplished using other methods.

The thickness of the cured adhesive layer formed from the adhesive composition should preferably be 1–100 μm. For DVD 9, for example, the thickness of the adhesive layer is set to 55 ± 15 μm in accordance with the standards described in the DVD Book.

The disc substrates coated with the adhesive composition should preferably be laminated under vacuum degassing. This approach is effective for rapid gas removal from the applied adhesive composition and for preventing gas bubbles from remaining between the substrates of the finished optical disc.

The optical disc of the present invention comprises disc substrates laminated by means of an adhesive, and at least one reflecting metallic film. As schematically shown in Figure 1, in its simplest aspect, the optical disc 10 of the present invention comprises two disc substrates 11, 11' of transparent plastic and a reflecting metallic film 12 made of a metal such as aluminum, for example, and formed on the signal-recording surface 11R of one of the disc substrates 11, yielding a structure in which the disc substrates 11, 11' are laminated through the agency of an adhesive layer 13 formed from the adhesive composition of the present invention, with the reflecting metallic film 12 disposed on the inside. Minute pits (not shown) formed in correspondence to recorded digital signals are present on the surface (signal-recording surface 11R) of the substrate 11 in contact with the reflecting metallic film 12, and the presence or absence of pits corresponding to recorded digital signals is read out by an object lens (not shown) by sensing the presence or absence of reflected light from the reflecting metallic film 12 as it is illuminated with readout laser light traveling in the direction of arrow A in the drawing. An optical disc of this aspect corresponds to the previously described type of disc in which data is read from a single layer on one side.

It will be apparent that the previously described type of optical disc in which data is read from a single layer on each side can be obtained by modifying the aspect in Figure 1, providing to substrate 11' pits and a reflecting metallic layer analogous to those provided to substrate 11, and illuminating substrate 11' with laser light directed from the direction opposite that indicated by A in the drawing.

Figure 2 depicts another aspect of the optical disc. The optical disc 20 pertaining to this aspect comprises two disc substrates 21, 21'; a semitransparent film 22 made of gold or the like and provided to one of the disc substrates 21; and an aluminum reflecting metallic film 24 on the other disc substrate 21'. The two disc substrates 21 and 21' are

laminated through the agency of an adhesive layer 25 formed from the adhesive composition of the present invention, with the semitransparent film 22 and the reflecting metallic film 24 disposed on the inside. In this aspect, pits (not shown) corresponding to recorded digital signals are formed on a signal-recording surface 21R in contact with the semitransparent film 22 of the substrate 21, and on the surface (signal-recording surface 25R) in contact with the reflecting metallic film 24 of the adhesive layer 25. Moving the object lens (not shown) of an optical head for reading recorded signals in the focusing direction (in the direction of propagation of laser light, indicated by A in the drawing) allows signals recorded on (signal-recording surface 21R) to be read by sensing light reflected from the semitransparent film 22, and signals recorded on (signal-recording surface 25R) to be read by sensing light reflected from the reflecting metallic film 24 following passage through the semitransparent film 22. Thus, this aspect allows signals on the two recording layers formed on the optical disc 20 to be read from one side. Specifically, the optical disc pertaining to this aspect corresponds to the previously described type of disc in which two layers are read from one side.

The optical disc of the present invention sustains very little corrosion of the metallic layers (particularly aluminum layers) when evaluated by means of durability tests under elevated temperature and high humidity conditions (80 °C, 90% relative humidity). When an adhesive used in the lamination of disc substrates contains traces of impurity ions or the like derived from starting materials or the like, the metallic reflecting layers must be provided with a protective coating because these impurities cause corrosion in aluminum and other thin metallic films. The adhesive composition of the present invention is characterized in that such thin metallic films experience very little corrosive action even in the presence of considerable amounts of impurity ions or other components introduced during the handling or synthesis of starting materials. In addition, an adhesive layer formed from the adhesive composition of the present invention, unlike a layer formed from a composition used as a common protective coating agent, can provide optical discs that are resistant to impact-induced breakage because this adhesive layer not only exhibits high adhesive power to aluminum, gold, polycarbonates, and various other materials used in the manufacture of optical discs, but also has high shock absorbing capabilities in relation to falls and other types of impact sustained by an optical disc. The adhesive composition of the present invention also satisfies the previously described

characteristic requirements for applications involving the bonding of substrates to optical discs.

Examples

The present invention will now be described in further detail through examples. It is apparent, however, that the present invention is not limited to these examples.

In these examples, the following acrylate oligomers, acrylate monomers, and photopolymerization initiators were used.

Oligomers

Oligomer A: Poly (tetramethylene glycol)-based urethane acrylate (UV-6100B, manufactured by The Nippon Synthetic Chemical Ind. Co., Ltd.)

Oligomer B: Polyester-based urethane acrylate (UV-3000B, manufactured by The Nippon Synthetic Chemical Ind. Co., Ltd.)

Oligomer C: Polycaprolactone-based urethane acrylate (UX-4101, manufactured by Nippon Kayaku Co., Ltd.)

Oligomer D: Propylene oxide-modified bisphenol A diacrylate (LIGHT ACRYLATE BP-4PA, manufactured by Kyoeisha Chemical Co. Ltd.)

Oligomer E: Polycarbonate-based urethane acrylate (UN-9200A, manufactured by Negami Chemical Industrial Co. Ltd.)

Monomers

HPA: 2-Hydroxypropyl acrylate (LIGHT ESTER HOP-A, manufactured by Kyoeisha Chemical Co. Ltd.)

PEA: Phenoxyethyl acrylate (BISUKOOTO [1] # 192, manufactured by Osaka Organic Chemical Ind.)

HBA: 4-Hydroxybutyl acrylate (4-hydroxybutyl acrylate manufactured by Mitsubishi Chemical Industries)

HPPA: 2-Hydroxy-3-phenoxypropyl acrylate (Epoxy Ester M-600A, manufactured by Kyoeisha Kagaku)

IBA: Isobornyl acrylate (LIGHT ACRYLATE IB-XA, manufactured by Kyoeisha Chemical Co., Ltd.)

Photopolymerization Initiator

D1173: Darocur 1173, manufactured by Ciba-Geigy

The adhesives in Examples 1-6 and Comparative Examples 1-9 formulated according to the compositions shown in Tables 1 and 2 (for example, the "oligomer A/HPA/D1173 = 80/20/1" in the composition of Example 1 shown in Table 1 refers to the action of mixing oligomer A, HPA, and D1173 in a weight ratio of 80:20:1) were applied by spin coating to the sputter film side of DVD 9 half discs (opaque discs obtained by sputtering polycarbonate substrates with aluminum, and semitransparent discs obtained by sputtering polycarbonate substrates with gold), the components were laminated using DLB-9900 (a DVD bonding apparatus manufactured by Sumitomo 3M), and the products were illuminated, on the side of the half disc provided with a thin metallic film, with ultraviolet light (for 20 seconds at 360 nm; UV illuminance: about 30 mW/cm^2) from a high-pressure mercury UV lamp to cure the adhesive.

The optical disc samples thus obtained were allowed to stand at room temperature for half a day or longer, placed in a 80 °C oven at 90% relative humidity, taken out 100, 250, or 500 hours later, inspected, and graded by durability. The grading results were represented as follows:

O: No particularly visible changes in aluminum film

Δ: Slight roughness of aluminum film noted

×: Considerable deterioration and penetration pitting of aluminum film noted

The grading results are shown in Tables 1 and 2.

Table 1

Example	Composition	Appearance 100 hours later	Appearance 250 hours later	Appearance 500 hours later
1	Oligomer A/HPA/D1173 = 80/20/1	O	O	O
2	Oligomer A/HPA/D1173 = 60/40/1	O	O	O
3	Oligomer A/HPA/D1173 = 40/60/1	O	O	O
4	Oligomer A/HPA/PEA/D1173 = 60/20/20/1	O	O	O
5	Oligomer A/HBA/D1173 = 50/50/1	O	O	O
6	Oligomer A/HPPA/D1173 = 50/50/1	O	O	O

Table 2

Comparative Example	Composition	Appearance 100 hours later	Appearance 250 hours later	Appearance 500 hours later
1	Oligomer A/HPA/D1173 = 30/70/1	O	O	Δ
2	Oligomer A/PEA/D1173 = 50/50/1	Δ	Δ	Δ
3	Oligomer A/IBA/D1173 = 50/50/1	Δ	Δ	×
4	Oligomer B/HPA/D1173 = 50/50/1	O	Δ	×
5	Oligomer C/HPA/D1173 = 60/40/1	×	×	×
6	Oligomer D/HPA/D1173 = 60/40/1	Δ	×	×
7	Oligomer E/IBA/D1173 = 60/40/1	Δ	Δ	×
8	HPA/D1173 = 100/1	×	×	×
9	Commercial UV-curing adhesive for DVDs	O	Δ	×
10	Commercial DVD9 disc	O	Δ	×

The tables indicate that accelerated durability tests performed for 500 hours at 80 °C and 90% relative humidity produced no visible changes in the aluminum films of discs obtained by bonding substrates with the adhesive compositions of Examples 1–6, which comprised a poly (tetramethylene glycol)-based urethane acrylate (oligomer A) and an acrylic monomer (HPA, HBA, HPPA) having hydroxyl groups, and in which the content of oligomer A constituted at least 40% of the total weight of the oligomer and acrylic monomer.

By contrast, 500-hour testing produced visible roughness in the aluminum films of discs obtained using the adhesive composition of Comparative Example 1, which comprised oligomer A and the acrylic monomer HPA and in which the content of oligomer A was less than 40% of the combined content of the oligomer and acrylic monomer. In addition, it took 250 hours or less for accelerated tests performed at 80 °C and 90% relative humidity to produce corrosion in the aluminum films of discs fabricated using adhesive compositions that contained poly (tetramethylene glycol)-based urethane acrylates (oligomers A) but were devoid of acrylic monomers having hydroxyl groups (as in Comparative Examples 2 and 3), compositions that contained acrylic monomers having hydroxyl groups but were devoid of polyether-based urethane acrylates (oligomers A) (as in Comparative Examples 4, 5, 6, and 8), or compositions that contained neither polyether-

based urethane acrylates (oligomers A) nor acrylic monomers having hydroxy groups, as in Comparative Example 7. Furthermore, the disc of Comparative Example 9, which was fabricated using a typical optical disc substrate adhesive available on the market, and the disc of Comparative Example 10, which was a commercially available DVD 9 product, were both found to have undergone corrosion over part of their aluminum films when visually inspected 250 hours after the start of the same accelerated durability test.

Thus, in accordance with the present invention, it is possible to use an adhesive composition that can ensure direct bonding of optical disc substrates and can control corrosion of a metallic film, particularly an aluminum film, in a durability test of a product optical disc performed for 500 hours under elevated temperature and high humidity conditions (80 °C and 90% relative humidity). Using this adhesive composition makes it possible to provide an optical disc having controlled corrosion of a metallic film under elevated temperature and high humidity.

The present invention is not limited to DVDs or other optical discs and can be adapted to magnetooptical (MO) discs and the like.

What is claimed is:

1. An adhesive composition comprising (1) a urethane acrylate having a poly(tetramethylene glycol) skeleton, (2) an acrylic ester having a hydroxyl group in its molecule, and (3) a photoinitiator, characterized in that the composition comprises 40% by weight or more of the urethane acrylate (1) based on the sum of weights of the urethane acrylate (1) and the acrylic ester (2).

2. The adhesive composition of claim 1, wherein the urethane acrylate (1) is an oligomer made up of, in addition to the poly (tetramethylene glycol) skeleton moiety, at least moieties derived from tetramethylxylene diisocyanate, hydroxyethyl acrylate, and ethylene glycol.

3. The adhesive composition of claim 1 or 2, wherein the acrylic ester (2) is one or more of hydroxyethyl acrylate, hydroxypropyl acrylate, hydroxybutyl acrylate, and hydroxyphenoxypropyl acrylate.

4. An optical disc comprising disc substrate bonded by an adhesive and at least one reflecting metallic film, characterized in that the adhesive is obtained by cure of the adhesive composition of any one of claims 1 to 3.

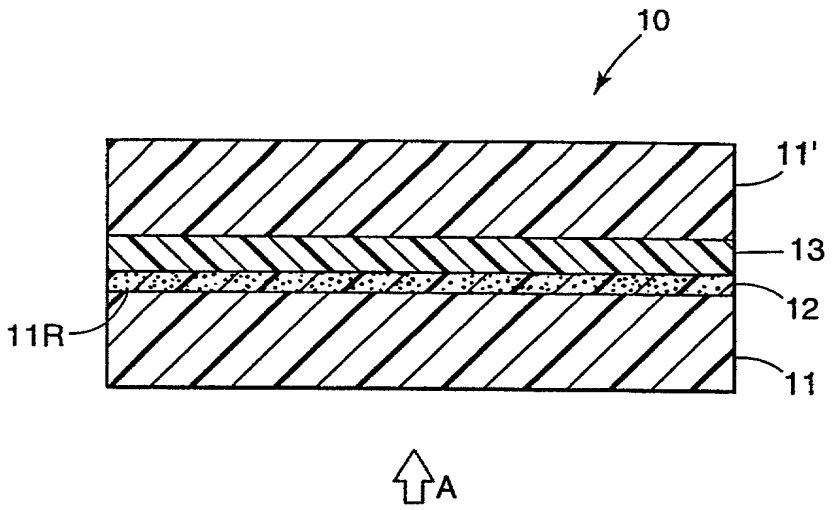


Fig. 1

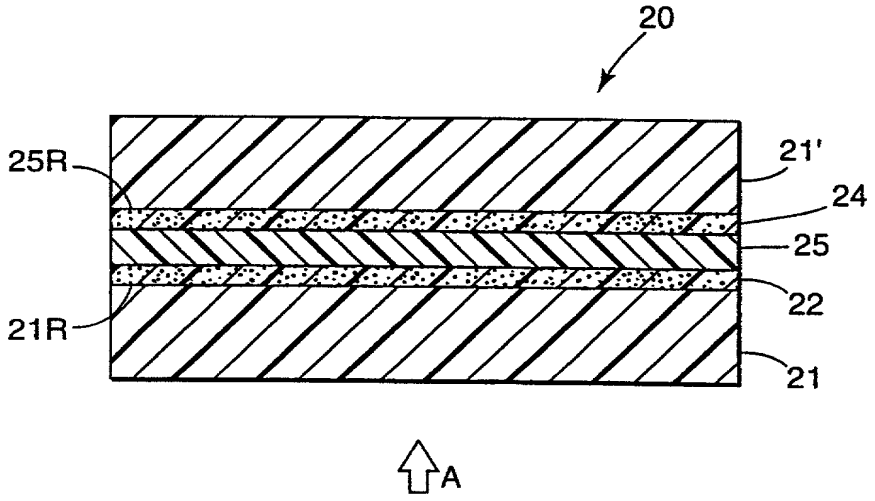


Fig. 2

Docket No. 54779US005

DECLARATION, POWER OF ATTORNEY, AND PETITION

I, a below named inventor, depose and say that: (1) my residence, citizenship, and mailing address are indicated below; (2) I have reviewed and understand the contents of my patent application, including the claims, as amended by any amendment specifically referred to herein, which is identified as PCT International Application No. PCT/US00/11422, filed April 27, 2000, bearing Attorney Docket Number 54779WO003; (3) I believe that I am the original, first, and sole inventor of the invention or discovery in

ADHESIVE COMPOSITION AND OPTICAL DISC USING THE SAME

described and claimed therein and for which a patent is sought; and (4) I hereby acknowledge my duty to disclose to the Patent and Trademark Office all information known to me to be material to the patentability as defined in Title 37, Code of Federal Regulations, §1.56*, and that no application for patent or inventor's certificate on this invention or discovery has been filed by me or my legal representatives or assigns in any country foreign to the United States of America except Japanese Application No. 11-160083, filed June 7, 1999 upon which I hereby claim foreign priority benefits under Title 35, United States Code Section 119.

I hereby appoint Gregory D. Allen (Reg. No. 35,048), Alan Ball (Reg. No. 42,286), Scott A. Bardell (Reg. No. 39,594), Carolyn A. Bates (Reg. No. 27,853), Bruce E. Black (Reg. No. 41,622), Colene E. H. Blank (Reg. No. 41,056), Jennie G. Boeder (Reg. No. 28,952), William J. Bond (Reg. No. 32,400), Arthur J. Brady (Reg. No. 42,356), Stephen W. Buckingham (Reg. No. 30,035), John A. Burtis (Reg. No. 39,924), Melissa E. Buss (Reg. No. 47,465), Gerald F. Chernivec (Reg. No. 26,537), James D. Christoff (Reg. No. 31,492), Philip Y. Dahl (Reg. No. 36,115), Janice L. Dowdall (Reg. No. 31,201), Lisa M. Fagan (Reg. No. 40,601), Carolyn A. Fischer (Reg. No. 39,091), Yen T. Florczak (Reg. No. 45,163), Darla P. Fonseca (Reg. No. 31,783), Melanie G. Gover (Reg. No. 41,793), Christopher D. Gram, (Reg. No. 43,643), Gary L. Griswold (Reg. No. 25,396), Doreen S. L. Gwin (Reg. No. 35,580), Michaele A. Hakamaki (Reg. No. 40,011), Karl G. Hanson (Reg. No. 32,900), Dean M. Harts (Reg. No. 47,634), Néstor F. Ho (Reg. No. 39,460), Rudolph P. Hofmann, Jr. (Reg. No. 38,187), Robert W. Hoke (Reg. No. 29,226), MarySusan Howard (Reg. No. 38,729), Stephen C. Jensen (Reg. No. 35,207), Robert H. Jordan (Reg. No. 31,973), Harold C. Knecht III (Reg. No. 35,576), Kent S. Kokko (Reg. No. 33,931), Andrew J. Leon (Reg. No. 46,869), Douglas B. Little (Reg. No. 28,439), Eloise J. Maki (Reg. No. 33,418), Michelle M. Michel (Reg. No. 33,968), William D. Miller (Reg. No. 37,988), Peter L. Olson (Reg. No. 35,308), Daniel R. Pastirik (Reg. No. 33,025), David B. Patchett (Reg. No. 39,326), Robert J. Pechman (Reg. No. 45,002), Carolyn V. Peters (Reg. No. 33,271), Scott R. Pribnow (Reg. No. 43,869), Ted K. Ringsred (Reg. No. 35,658), Steven E. Skolnick (Reg. No. 33,789), Robert W. Sprague (Reg. No. 30,497), Brian E. Szymanski (Reg. No. 39,523), James J. Trussell (Reg. No. 37,251), Lucy C. Weiss (Reg. No. 32,834), Bradford B. Wright (Reg. No. 34,459), and Kimberly S. Zillig (Reg. No. 46,346) my attorneys and/or agents with full powers (including the powers of appointment, substitution, and revocation) to prosecute this application and any division, continuation, continuation-in-part, reexamination, or reissue thereof, and to transact all business in the Patent and Trademark Office connected therewith; the mailing address and the telephone number of the above-mentioned attorneys and/or agents are

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The undersigned petitioner declares further that all statements made herein of his own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

*Title 37, Code of Federal Regulations, §1.56 is attached.

§1.56 Duty to disclose information material to patentability.

(a) A patent by its very nature is affected with a public interest. The public interest is best served, and the most effective patent examination occurs when, at the time an application is being examined, the Office is aware of and evaluates the teachings of all information material to patentability. Each individual associated with the filing and prosecution of a patent application has a duty of candor and good faith in dealing with the Office, which includes a duty to disclose to the Office all information known to that individual to be material to patentability as defined in this section. The duty to disclose information exists with respect to each pending claim until the claim is cancelled or withdrawn from consideration, or the application becomes abandoned. Information material to the patentability of a claim that is cancelled or withdrawn from consideration need not be submitted if the information is not material to the patentability of any claim remaining under consideration in the application. There is no duty to submit information which is not material to the patentability of any existing claim. The duty to disclose all information known to be material to patentability is deemed to be satisfied if all information known to be material to patentability of any claim issued in a patent was cited by the Office or submitted to the Office in the manner prescribed by §§ 1.97(b)-(d) and 1.98. However, no patent will be granted on an application in connection with which fraud on the Office was practiced or attempted or the duty of disclosure was violated through bad faith or intentional misconduct. The Office encourages applicants to carefully examine:

(1) prior art cited in search reports of a foreign patent office in a counterpart application, and

(2) the closest information over which individuals associated with the filing or prosecution of a patent application believe any pending claim patentably defines, to make sure that any material information contained therein is disclosed to the Office.

(b) Under this section, information is material to patentability when it is not cumulative to information already of record or being made of record in the application, and

(1) It establishes, by itself or in combination with other information, a prima facie case of unpatentability of a claim; or

(2) It refutes, or is inconsistent with, a position the applicant takes in:

(i) Opposing an argument of unpatentability relied on by the Office, or

(ii) Asserting an argument of patentability.

A prima facie case of unpatentability is established when the information compels a conclusion that a claim is unpatentable under the preponderance of evidence, burden-of-proof standard, giving each term in the claim its broadest reasonable construction consistent with the specification, and before any consideration is given to evidence which may be submitted in an attempt to establish a contrary conclusion of patentability.

(c) Individuals associated with the filing or prosecution of a patent application within the meaning of this section are:

(1) Each inventor named in the application;

(2) Each attorney or agent who prepares or prosecutes the application; and

(3) Every other person who is substantively involved in the preparation or prosecution of the application and who is associated with the inventor, with the assignee or with anyone to whom there is an obligation to assign the application.

(d) Individuals other than the attorney, agent or inventor may comply with this section by disclosing information to the attorney, agent, or inventor.

Wherefore, I pray for grant of Letters Patent for the invention or discovery described and claimed in the aforementioned specification and I hereby subscribe my name to the foregoing specification and claims, declaration, power of attorney, and this petition, on the day set forth below.

1-D SANA FUJII 10/24/01

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